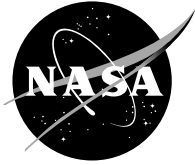


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Elevated Temperature Fatigue Endurance of Three Ceramic Matrix Composites

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Abstract

High-cycle fatigue endurance of three candidate materials for the acoustic liners of the Enabling Propulsion Materials' Nozzle Program was investigated. The ceramic matrix composite materials investigated were N720/AS (Nextel 720, 3M Corporation), Sylramic S200 (Dow Corning), and UT-22. High-cycle fatigue tests were conducted in air at 910 °C on as-machined specimens and on specimens subjected to tensile cyclic load excursions every 160 hr followed by thermal exposure at 910 °C in a furnace up to total exposure times of 2066 and 4000 hr. All the fatigue tests were conducted in air at 100 Hz with a servohydraulic test machine. In the as-machined condition, among the three materials investigated only the Sylramic S200 exhibited a deterministic type of high-cycle fatigue behavior. Both the N720/AS and UT-22 exhibited significant scatter in the experimentally observed high-cycle fatigue lives. Among the thermally exposed specimens, N720/AS and Sylramic S200 materials exhibited a reduction in the high-cycle fatigue lives, particularly at the exposure time of 4000 hr.

Introduction

The objective of the investigation was to evaluate the high-cycle fatigue endurance of the three candidate ceramic matrix composites (CMCs) to facilitate the material down selection process for the exhaust nozzle acoustic liners in the Enabling Propulsion Materials (EPM) program (ref. 1). Acoustic liners fabricated with CMCs were necessary to achieve the noise reduction requirement under the severe thermal-acoustic environment while meeting the constraint on overall weight of the nozzle. The candidate CMCs considered for acoustic liners were (1) General Electric's Oxide/Oxide (N720/AS), (2) Dow Corning's Sylramic S200 composite, and (3) UT-22 fabricated by United Technologies Research Center (UTRC). Selection of all the CMCs for the investigation was based on the material properties of these materials satisfying certain minimum design guidelines (ref. 2). Detailed investigations on the fabricability and mechanical properties of these materials are available in reference 2. In this report, the testing techniques used to perform high temperature, high frequency fatigue tests on these three materials and high-cycle fatigue data generated are documented. The fatigue endurance of the three CMCs is compared with maximum stress-based fatigue life relationships.

Materials and Specimens

The oxide/oxide system consisted of a fabric woven from N720 fiber tows and impregnated with an aluminosilicate (glass-ceramic) matrix. The processing involved both autoclave and firing steps to achieve the desired composition for the material. Examples of two types of acoustic liners (flat and rib-stiffened) produced with this fabrication technique were discussed in reference 2. Basic processing steps and mechanical properties of oxide/oxide CMCs were reported by Jurf and Butner (ref. 3). In particular,

John et al. (ref. 4) discussed the notch-sensitivity of a similar oxide/oxide material. In the N720/AS CMC investigated by John et al. (ref. 4), fabrication of the material resulted in a weak bond between the fibers and the matrix and no engineered interface existed. The N720/AS composite investigated in this study contained 12 plies with a nominal thickness of 3.0 mm.

The Sylramic S200 composite was manufactured with Ceramic Grade (CG) Nicalon (ATK Composite Optics, Inc.) fabric woven to an 8 harness stain-weave configuration. The fabric was subsequently coated with a proprietary interface by Dow Corning. Typical interface materials used for this class of CMCs include carbon (C) and boron nitride (BN) (refs. 5 to 7). The coated fabric was impregnated with a preceramic polymer and cured in an autoclave with well-established lay-up and vacuum processing techniques. A cross-ply lay up $[0/90]_{4S}$ was used for fabricating the material used in this study. The preceramic polymer was converted into Si-N-C matrix by pyrolyzing the cured composite in an inert furnace. Several polymer impregnation and pyrolysis (PIP) cycles were used to achieve the required density for the composite. Additional details on the PIP method for manufacturing CMCs are available in reference 8. The nominal thickness of Sylramic S200 composite was 3.0 mm.

The UT-22 composite consisted of barium aluminosilicate (glass-ceramic) matrix reinforced with CG Nicalon fibers. Coatings of BN and silicon carbide (SiC) were applied in that order to the fibers by chemical vapor deposition to provide a weak interfacial zone for crack deflection and to prevent the interdiffusion of BN into the matrix, respectively. Unidirectional prepreg tape was prepared by drawing the dual coated CG Nicalon tow through slurry consisting of glass powder, organic binder, and water and winding it on a hexagonal drum. Composite was manufactured by sequentially implementing prepreg lay-up followed by thermal decomposition of the binder to develop the preform and finally hot pressing the preform at elevated temperature and pressure (ref. 2). The UT-22 material used in this study had a cross-ply lay up of $[0/90]_{6S}$ and a nominal thickness of 2.7 mm. More details on the panel sizes and number of UT-22 panels manufactured by UTRC are available in reference 2.

The test specimens used in this investigation had a uniform gage section with a width of 10.2 mm, grip ends that were 12.7 mm wide, and an overall length of 152 mm (ref. 9). Test specimens were machined from N720/AS and Sylramic S200 composite plates with diamond-tipped tools. Laser machining was used to machine the test specimens from the UT-22 plates (ref. 2).

High-Cycle Fatigue Testing

High-cycle fatigue tests on specimens from all the three materials were conducted in air at 910 °C. This temperature was selected based on the anticipated hot-side temperature of the acoustic liner in the design analyses. Tensile properties (elastic modulus, Poisson's ratio, and ultimate tensile strength (UTS)) for the three CMCs at 910 °C are listed in table 1. A servohydraulic test system, equipped with water-cooled wedge grips and a custom fabricated SiC susceptor for heating the specimens, was used for performing the tests. Further details on the fabrication and usage of the susceptor are available in reference 9. Two R-type beaded thermocouples, located inside the susceptor on either side of the test specimen, were used to control and monitor the temperature of the test specimen during the fatigue test. A 5 kW induction heating unit was used to heat the specimen located in the susceptor. The test system

TABLE 1.—TENSILE PROPERTIES OF THE THREE
CMCS AT 910 °C IN AIR (REF. 2)

Material	Elastic modulus (E_{11}), GPa	Poisson's ratio (ν_{12})	UTS, MPa
N720/AS	73.8	0.093	139
Sylramic S200	98.6	0.093	226
UT-22	124	0.28	147

alignment was performed with a strain-gauged, rectangular cross-section bar. The test rig was aligned such that the maximum bending strain contribution was no greater than 5 percent at an axial load of 4.4 kN. All the high-cycle fatigue tests were conducted at 100 Hz with a sinusoidal waveform and $R = \sigma_{min} / \sigma_{max} = 0.1$. The gripping pressure used for the three types of CMC specimens was 2,070 kPa. At this pressure level, N720/AS specimens failed in the vicinity of the gripping areas. To avoid such failures, the gripping pressure for these specimens was reduced to 1,380 kPa. In addition, soft, light-weight, and corrosion-resistant aluminum wire mesh (0.41 mm wire diameter and 1.2 mm width opening) was used as a tab material to evenly distribute the gripping force for the oxide/oxide specimens. Usage of the soft aluminum mesh tabs around the grip ends and reduction of the gripping pressure eliminated failures near the gripping regions for the as-machined oxide/oxide specimens.

High-cycle fatigue tests were conducted on as-machined specimens and on specimens subjected to tensile cyclic load excursion at room temperature followed by thermal exposure in furnace for all the three CMCs. The latter specimens were initially subjected to a tensile stress of 103 MPa at room temperature and subsequently exposed to 910 °C in a furnace. After every 160 hr, these specimens were subjected to the same tensile stress at room temperature. A tensile stress of 103 MPa was selected because it was higher than the proportional limit strengths of all three materials investigated. Because the selected tensile stress was greater than the proportional limit strength, the tensile excursion could potentially damage the composite (for example, cracking within the matrix) and the subsequent high temperature treatment was designed to further expose any such damaged material to environmental influence. The objective of the program was to investigate the debit, if any, in the HCF lives of the CMCs due to prior mechanical loads and thermal exposures. The previously described mechanical loading and thermal exposure cycle was repeated on the test specimens until the required thermal exposure durations (2066 and 4000 hr) were achieved (ref. 2). In the high-cycle fatigue tests, if a CMC specimen did not fail after 10 million cycles, the test was considered a runout.

Results and Discussion

The high-cycle fatigue data generated on the three CMCs are listed in tables 2 to 4. These tables contain fatigue data generated on the as-machined specimens and those subjected to cyclic tensile load excursions and thermal exposure. A power-law type fatigue life relationship based on the maximum stress in a cycle was used to characterize the data generated with the as-machined specimens for the three CMCs.

TABLE 2.—HIGH-CYCLE FATIGUE DATA
OF N720/AS AT 910 °C ($R = 0.1$)

Specimen number	Thermal exposure, hr	σ_{max} MPa	N_f cycles	Failure location ^d
5079-8	-----	138	3 700	Gage
5079-9	-----	131	11 200	Gage
5079-12	-----	124	23 300	Transition
5079-6	-----	124	^a 10 000 000	N/A ^b
5042B-4	-----	121	25 300	Grip ^c
5079-13	-----	117	^a 10 000 000	N/A ^b
5079-5	-----	110	^a 10 000 000	N/A ^b
5042B-6	-----	103	^a 10 000 000	N/A ^b
5042A-10	2066	124	43 700	Grip
5042A-12	2066	103	^a 10 000 000	N/A ^b
5045B-8	4000	103	20 200	Trans./Grip

^aRunout.

^bN/A: not applicable.

^cA higher grip pressure of 2 070 kPa was used for this test.

^dFor the rest of the tests a grip pressure of 1 380 kPa was used.

TABLE 3.—HIGH-CYCLE FATIGUE DATA OF SYLRAMIC S200 AT
910 °C ($R = 0.1$)

Specimen number	Thermal exposure, hr	σ_{max} , MPa	N_f cycles	Failure location
147-10	0	138	1 109 900	Transition
149-16	0	138	1 374 600	Gage
147-14	0	121	1 456 600	Gage
147-4	0	121	2 508 400	Gage
147-12	0	103	2 918 900	Transition
147-6	0	103	5 305 600	Transition
147-8	0	86	^a 10 000 000	N/A ^b
147-21	0	86	^a 10 000 000	N/A ^b
147-19	2066	103	6 229 200	Transition
147-20	2066	86	^a 10 000 000	N/A ^b
147-17	4000	103	375 600	Transition
147-16	4000	86	^a 10 024 700	N/A ^b

^aRunout.

^bN/A: not applicable.

TABLE 4.—HIGH-CYCLE FATIGUE DATA OF UT-22 AT 910 °C ($R = 0.1$)

Specimen number	Thermal exposure, hr	σ_{max} , location	N_f , hr	Failure, MPa
2-96-G1	0	121	462 700	Transition
2-96-G4	0	110	1 134 700	Transition
42-95-G21 ^a	0	103	16 500	Gage
2-96-G2	0	103	2 011 300	Transition
54-95-G30	0	97	8 279 300	Gage
42-95-G26 ^a	0	90	11 700	Gage
42-95-G22 ^a	0	86	2 098 500	Transition
2-96-G3	0	86	5 765 600	Gage
54-95-G31	0	83	^b 10 000 000	N/A ^c
42-95-G27 ^a	0	83	^b 10 000 000	N/A ^c
42-95-G25 ^a	0	76	^b 10 000 000	N/A ^c
42-95-G23 ^a	0	69	^b 10 000 000	N/A ^c
42-95-G20 ^a	0	66	...	Gage/Tr. ^d
40-95-G7	2066	103	386 000	Gage
40-95-G8	2066	86	2 504 900	Transition
40-95-G10	4000	103	837 000	Gage
40-95-G11	4000	86	7 199 400	Gage

^aUTRC determined that UT-22 panel 42-95 had low flexural strength due to fabrication difficulties associated with scale-up of the size of composite panels (ref. 2).

^bRunout.

^cN/A: not applicable.

^dSpecimen failed during load-up at 66 MPa.

$$\sigma_{max} = B(N_f)^b$$

where, σ_{max} is the maximum stress in MPa, N_f is the number of cycles to failure, B is the coefficient, and b is the exponent. For all the CMCs, the runout data as well as the fatigue datum corresponding to specimen failure in the grip region were omitted while computing the life relationships. In the case of UT-22, data points corresponding to the low strength panel (specimen numbers beginning with 42-95-xxx in table 4) were also not considered in establishing the life relationship for that material. Difficulties associated with processing during fabrication of the UT-22 panels at UTRC (ref. 2) led to the low strength of panel 42-95.

However, UTRC identified the issues associated with the low strength of the panels and subsequently fabricated acceptable panels. High-cycle fatigue data generated and the fatigue life relationships calculated from regression analyses are shown in figures 1 to 3 for all the three CMC materials. The number of data points used in each regression analysis and coefficients and exponents for the fatigue life relationships of the CMCs are listed in table 5.

TABLE 5.—CONSTANTS FOR MAXIMUM STRESS-FATIGUE LIFE RELATIONSHIPS

Material	Environment, hr	Exposure time	n^a	b	B , MPa
N720/AS	Air	---	3	-0.0583	224
Sylramic S200	Air	---	6	-0.248	4400
UT-22	Air	---	5	-0.121	602

^aNumber of data points used in the regression analysis to determine b and B in the fatigue life relationship.

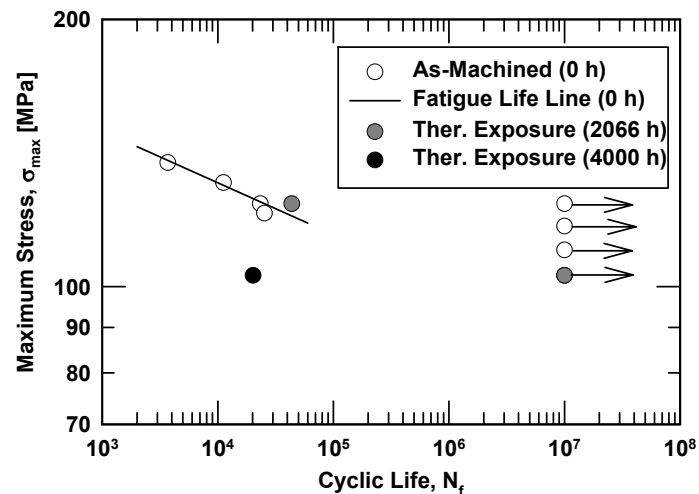


Figure 1.—High-cycle fatigue behavior of N720/AS at 910 °C.

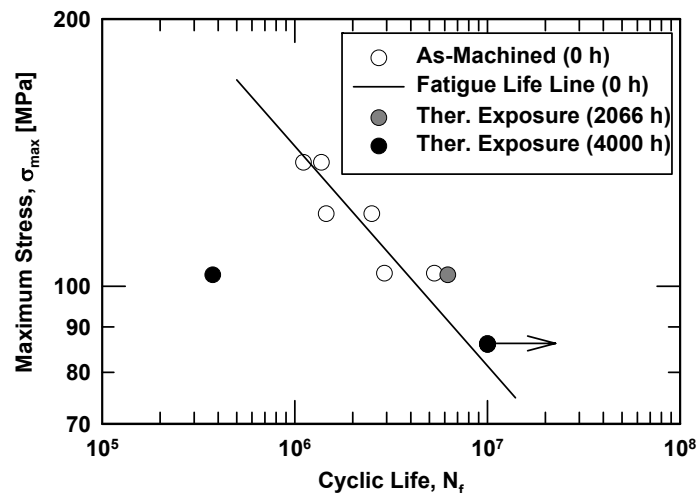


Figure 2.—High-cycle fatigue behavior of Sylramic S200 at 910 °C.

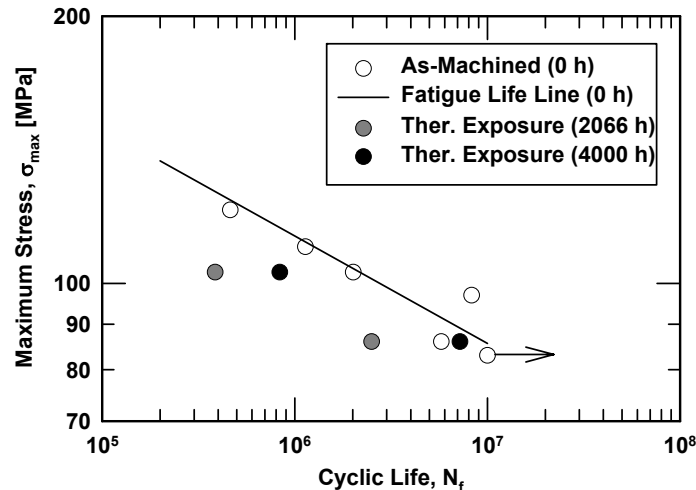


Figure 3.—High-cycle fatigue behavior of UT-22 at 910 °C.

The fatigue life relationship of N720/AS exhibited relatively shallow slope ($b = -0.0583$; table 5) and a limited “deterministic” life regime. Similar fatigue behavior was reported for a brittle matrix composite (graphite fiber reinforced glass) by Talreja (ref. 10). The fatigue failure process in N720/AS composite appears to be dominated by the strength of the woven fiber cloth. It is speculated that typically the test specimen did not fail before 10 million cycles, if the applied maximum stress is lower than the strength of the woven fiber cloth. Any applied stress in the vicinity of the strength of the woven fiber cloth most likely produced failure of the specimen in a few cycles (table 5). The slope of Sylramic S200 composite’s fatigue life relationship was relatively steep ($b = -0.248$; table 5) and fatigue life data for a given maximum stress exhibited only “nominal” (typically a factor of two) scatter. Fractographic investigations performed earlier on fatigued specimens (ref. 11) revealed that cracks primarily initiated at the corners of the rectangular cross-section Sylramic S200 specimens and within the matrix rich regions at the edges of the specimens during high-cycle fatigue loading. As mentioned earlier in this report, for UT-22 the high-cycle fatigue data generated from the low-strength panels are not shown in figure 3. However, fatigue data generated on both the low- and high-strength UT-22 panels are included in Table 4 for completeness. In the case of UT-22 composite, the fatigue life relationship calculated with the data generated from the high-strength panels essentially establishes an upper bound for all the high-cycle fatigue data generated on this composite (fig. 3). Slope of the UT-22 composite’s fatigue life relationship ($b = -0.121$; table 5) was in between those observed for the other two CMCs investigated in this study.

High-cycle fatigue data generated at 910 °C on specimens subjected to tensile cyclic load excursions followed by thermal exposure are also included in tables 2 to 4 and in figures 1 to 3 for the three composite materials. No significant differences in the high-cycle fatigue lives were observed between the as-machined and 2,066 hr thermal exposure conditions for N720/AS (fig. 1). However, the high-cycle fatigue life of N720/AS was reduced significantly at a thermal exposure time of 4,000 hr. Based on the limited amount of data generated in this study, it appears that the fatigue durability of N720/AS composite degrades due to thermal exposure beyond 2,000 hr. The high-cycle fatigue life Sylramic S200 composite was also not significantly influenced by 2,066 hr thermal exposure (fig. 2). After 4,000 hr thermal exposure, substantial reduction in the fatigue life was observed only at the higher maximum tensile stress of 103 MPa (table 3). This observation implies that the influence of thermal exposure on the fatigue life of Sylramic S200 composite is a function of the maximum stress in high-cycle fatigue loading. In the case of UT-22 composite, fatigue lives of the specimens subjected to 2,066 hr thermal exposure were lower than those from the specimens subjected to 4,000 hr thermal exposure (fig. 3). This observed trend is different from that observed for N720/AS and Sylramic S200 and reasons for this

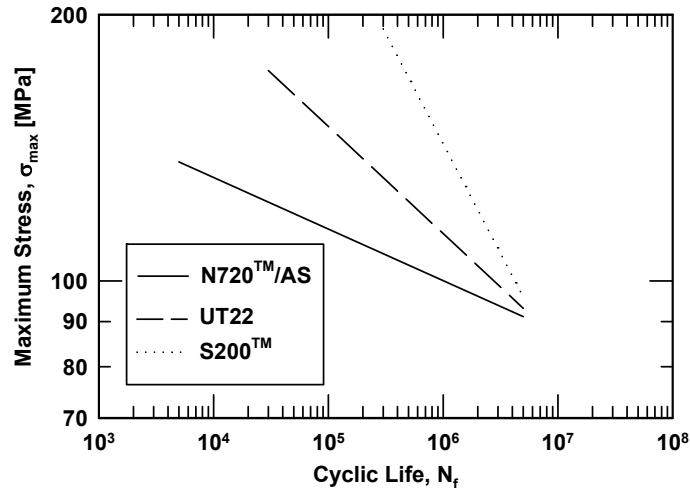


Figure 4.—Comparison of high-cycle fatigue life relationships at 910 °C.

unusual behavior are not completely evident from the current study. Scatter inherent in the fatigue data might be a possibility. In order to resolve these issues with any certainty, multiple fatigue tests at a given stress level are required on specimens subjected to different thermal exposure times.

The fatigue life relationships calculated from the data generated on as-machined specimens are compared for the three CMCs in figure 4. It is evident from this plot that at 910 °C for a given maximum stress the highest fatigue durability in the high-cycle regime (10^5 to 10^7 cycles) is exhibited by the Sylramic S200, followed by UT-22 and N720/AS. This observation and a series of other design criteria were used to down select Sylramic S200 composite for further evaluation as the acoustic liner material in the EPM program (ref. 2).

Concluding Remarks

Elevated temperature, high-cycle fatigue endurance of three ceramic matrix composite materials, N720/AS, Sylramic S200, and UT-22, was evaluated in air to facilitate the material down selection process for the acoustic liners in the EPM program. High frequency fatigue tests were conducted at 910 °C on both as-machined specimens and on specimens subjected to tensile cyclic load excursions at room temperature followed by furnace exposures at 910 °C up to total exposure times of 2,066 and 4,000 hr. Among the three materials investigated in the as-machined condition, the highest fatigue endurance and deterministic behavior was exhibited by the Sylramic S200 composite, followed by UT-22 and N720/AS. All the three materials exhibited degradation of high-cycle fatigue life to some degree due to prior tensile cyclic load excursions followed by thermal exposures. From the high-cycle fatigue durability viewpoint, the results from this investigation identified Sylramic S200 composite as the material of choice for the acoustic liners in the EPM program.

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